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(54) Process for preparing sodium percarbonate.

(57) A process for preparing sodium percarbonate, $2Na_2CO_3 \cdot 3H_2O_2$, starting from sodium carbonate and hydrogen peroxide having a H_2O_2 concentration of from 40 to 80% by weight.

The reaction takes place in the presence of a percarbonate stabilizer, for example magnesium silicate, and under reduced pressure. The reduced pressure and feeding rate of hydrogen peroxide to the reactor containing sodium carbonate are adjusted so as to maintain the reaction mass at a temperature from 20° to 40°C. The product is then dried.

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"PROCESS FOR PREPARING SODIUM PERCARBONATE"

The present invention relates to a process for preparing sodium percarbonate, $2Na_2CO_3 \cdot 3H_2O_2$.

As is known, sodium percarbonate may be prepared by reacting, at ambient pressure, sodium carbonate with an hydrogen peroxide solution. The reaction heat is removed from the reactor by outside cooling in order to prevent the temperature from rising, which could result in decomposition of the product. The product is then dried.

According to this process, the hydrogen peroxide solution must be fed slowly and gradually to the reactor containing sodium carbonate in order to avoid overheating of the product. As a consequence, the reaction times are long. Furthermore it is necessary to use concentrated hydrogen peroxide to minimize product decomposition phenomena, the product stability decreasing as the water amount increases. Moreover, the resulting product possesses a rather high brittleness.

The present invention accordingly aims to provide a process for preparing sodium percarbonate which does not require either cooling or heating and permits an easy control of the temperature of the mass being reacted, whereby it is possible to employ short reaction times while obtaining a high reactor productivity. It is also

desired to be able to utilize, optionally, a relatively dilute hydrogen peroxide solution. It is further desired to be able to obtain a product having a low brittleness, with a high content of active oxygen and a good stability.

The present invention provides a process for preparing sodium percarbonate, according to which sodium carbonate and an aqueous solution of hydrogen peroxide having a H_2O_2 concentration of from 40 to 80% by weight are reacted in the presence of a stabilizer of percarbonate, and the product is then dried. The process is characterized in that the reaction is conducted under reduced pressure and in that the reduced pressure and the feeding rate of the hydrogen peroxide solution to the reactor containing the sodium carbonate are adjusted so as to maintain the reaction mass at a temperature from 20 to 40°C.

The process according to the invention is generally accomplished under substantially adiabatic conditions since the reaction heat is eliminated, due to the reduced pressure, as evaporation heat of the water introduced into the reaction system through the hydrogen peroxide solution. After having fixed the temperature range in which the process will be operated, a reduced pressure is created such as to ensure the water evaporation at these temperatures, whereupon the hydrogen peroxide solution is introduced at such a rate as not to exceed the pre-assigned temperature values.

The reaction temperature ranges from 20 to 40°C. When operating below 20°C, the reaction times tend to be too long, while when the process is operated above 40°C sodium percarbonate tends to decompose. Preferably the process is operated at temperatures ranging from 25 to 35°C.

The residual pressure does not generally exceed 60 mm of Hg; usually the residual pressure is from 10 to 30 mm of Hg.

The H_2O_2 concentration of the hydrogen peroxide solution ranges from 40 to 80% by weight. When the process is operated at an H_2O_2 concentration below 40%, the reaction times are too long and the product may decompose during the reaction step. Preferably, a hydrogen peroxide solution at a H_2O_2 concentration of from 60 to 70% is used.

The starting sodium carbonate may be, for example, anhydrous Na_2CO_3 or $Na_2CO_3.H_2O$. Preferably, anhydrous Na_2CO_3 is employed.

Drying of the product is generally carried out at temperatures ranging from 40 to 60°C. The drying may take place under static conditions, for example in a chamber through which a hot air stream is made to flow, or under dynamic conditions, for example in a fluidized bed.

As sodium percarbonate stabilizers it is possible to utilize substances already known for such purposes such as, for example, magnesium salts, alkaline salts of polycarboxylic acids, sodium phosphonates, sodium hexametaphosphate, sodium pyrophosphate, sodium orthosilicate and sodium metasilicate. Particularly satisfactory results are obtained by using magnesium silicate.

The amount of stabilizer may range, for example, from 0.5 to 5 g per mole of sodium percarbonate.

The molar ratio between H_2O_2 and Na_2CO_3 is generally from 0.8 to 1.8. However, if the process is operated with molar ratios lower than the stoichiometric

value of 1.5, the active oxygen content of the product is rather low, although still sufficient to allow the utilization of the product in detergent formulations. If the process is operated with a molar ratio higher than 1.8, there is a considerable loss of H_2O_2. Preferably the process is operated with a molar ratio ranging from 1.6 to 1.7.

To obtain a good granulometry of percarbonate it is preferable to use a granular sodium carbonate having a small amount of product with a particle size exceeding 0.8 mm of mesh size of the screen and a small amount of product with a particle size below 0.16 mm of mesh size of the screen (the mesh size of the screen is the distance between two parallel and subsequent wires).

The reaction may be conducted in a mixer capable of ensuring intimate contact between the reagents, and which is vacuum tight.

After drying, the product is screened to remove the coarsest fraction (generally the one above 0.8 mm of mesh size of the screen) as well as the finest fraction (generally the one below 0.16 mm of mesh size of the screen). The coarsest fraction can be ground and recycled, along with the finest one, to the reaction step. This product recycle does not impair either the accomplishment of the process or the granulometry of the final product.

The resulting product exhibits a granular morphology with a smooth surface. Its content of active oxygen is generally equal to or higher than 14% by weight. The chemical yield, calculated on the basis of sodium carbonate, generally ranges from 85 to 95%.

The invention will be further described with reference to the following illustrative Examples.

EXAMPLE 1

Starting from granular anhydrous 99.5% Na_2CO_3, the fraction having a particle size above 0.8 mm was separated by screening and, after grinding, was returned to the remaining portion of the product.

2,100 g of Na_2CO_3, 1,100 g of recycled sodium percarbonate and 53 g of colloidal magnesium silicate were introduced into a reactor. The reactor was a helical double-ribbon mixer, type "Ribbon Mixer.". The reactor jacket was heated, at the beginning of the operation, by means of water circulating at 32-33°C, in order to avoid heat dispersion. Vacuum was applied and 1,764 g of hydrogen peroxide solution having an H_2O_2 concentration of 64% by weight were sucked into the equipment. The H_2O_2/Na_2CO_3 molar ratio was therefore equal to 1.69.

Feeding occurred in 25 minutes, under a residual pressure of about 20-25 mm of Hg. The reaction mass temperature was from 28 to 33°C. On completion of feeding, the mass was kept under stirring, always under reduced pressure, for a further 10 minutes.

The product, having a residual moisture of about 10%, was directly transferred into a fluidized bed drier where it was dried, by means of air heated to 44-46°C, for a time period of 15 minutes.

The dry product was screened on screens having a mesh size of 0.8 mm and 0.15 mm. The coarse product was ground and recycled, along with the fine product, to a successive operative step.

By deducting the recycled percarbonate, 3,020 g of dry product were obtained. The operative yield, calculated on the basis of Na_2CO_3, was 90%.

The product exhibited the following characteristics :

active oxygen titre	: 14.1	%
sodium percarbonate titre	: 92.2	%
bulk density	: 1.028	g/cm^3
brittleness	: 6.8	%
stability in dry conditions	: 94.5	% .

Brittleness was determined on the fraction of 0.4 - 0.8 mm by the test of the European Laboratory Standard Practice Instructions, No. 7/17050 of June 10, 1971.

Stability in dry conditions was determined after an eighteen-day residence time in an oven at 37°C, at a relative humidity of 70%. The product granules were round, smooth and compact.

The product granulometry was as follows :

above	0.8	mm of mesh size	:	3.6%
from	0.4	to 0.8 mm	:	60.2%
from	0.2	to 0.4 mm	:	22.2%
from	0.16	to 0.2 mm	:	12.0%
below	0.16	mm	:	2.0% .

EXAMPLE 2

The procedure described in Example 1 was followed, except for the following.

- sodium carbonate was used without carrying out any screening and grinding of the coarse product;
- the hydrogen peroxide titre was 69.5% of H_2O_2;

- the H_2O_2/Na_2CO_3 molar ratio was 1.66;
- the amount of recycled sodium percarbonate was 900 g;
- feeding of hydrogen peroxide occurred in 23 minutes;
- the residual pressure was 17 - 22 mm of Hg;
- the temperature of the reaction mass was from 23 to 32°C.

The yield calculated on the basis of Na_2CO_3 amounted to 88.2%. The product obtained had an active oxygen titre of 14.0% and a brittleness of 6.6%.

EXAMPLE 3

The procedure described in Example 1 was followed, except for the following :

- the hydrogen peroxide titre was 59.6% of H_2O_2;
- the H_2O_2/Na_2CO_3 molar ratio was 1.77;
- the amount of recycled sodium percarbonate was 900 g;
- feeding of hydrogen peroxide occurred in 40 minutes;
- the residual pressure was 10 - 20 mm of Hg;
- the temperature of the reaction mass was from 24 to to 29°C.

The yield calculated on the basis of Na_2CO_3 amounted to 94.6%. The product obtained had an active oxygen titre of 14.6% and a brittleness of 12%.

C L A I M S :

1. A process for preparing sodium percarbonate by reacting, in the presence of a stabilizer of the percarbonate, sodium carbonate and an hydrogen peroxide solution having a H_2O_2 concentration of from 40 to 80% by weight, and by drying the product, characterized in that the reaction is conducted under reduced pressure and in that the reduced pressure as well as the feed rate of the hydrogen peroxide solution to the reactor containing the sodium carbonate are adjusted so as to maintain the reaction mass at a temperature from 20° to 40°C.

2. A process as claimed in Claim 1, characterized in that the reaction mass is maintained at a temperature from 25° to 35°C.

3. A process as claimed in Claim 1 or 2, characterized in that anhydrous sodium carbonate is used.

4. A process as claimed in any of Claims 1 to 3, characterized in that the H_2O_2 concentration of the hydrogen peroxide solution is from 60 to 70%.

5. A process as claimed in any of Claims 1 to 4, characterized in that the H_2O_2/Na_2CO_3 molar ratio is from 0.8 to 1.8.

6. A process as claimed in Claim 5, characterized in that the H_2O_2/Na_2CO_3 molar ratio is from 1.6 to 1.7.

7. A process as claimed in any of Claims 1 to 6, characterized in that the stabilizer of sodium percarbonate is magnesium silicate.

8. Sodium percarbonate obtained according to the process as claimed in any of Claims 1 to 7.

European Patent Office

EUROPEAN SEARCH REPORT

0070711
Application number

EP 82 30 3753.6

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim
X	DE - A1 - 2 631 917 (FMC CORP.) * claim 1 *	1,2,4
A	DE - C - 425 598 (DEGUSSA)	7
A	US - A - 3 870 783 (R.E. HALL et al.)	

CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)

C 01 B 15/10

TECHNICAL FIELDS SEARCHED (Int.Cl.³)

C 01 B 15/00

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same category
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: earlier patent document, but published on, or after the filing date
D: document cited in the application
L: document cited for other reasons

&: member of the same patent family, corresponding document

X The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Berlin	31-08-1982	KESTEN

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